"PREPARATION AND POST-MODIFICATION OF METALLO-SILICATE MATERIALS SYNTHESIZED FROM AGRO-INDUSTRIAL WASTES AND THEIR PHYSICO-CHEMICAL APPLICATIONS"

Pre-Synopsis

Submitting for the Degree of

DOCTOR OF PHILOSOPHY IN PHYSICS

To the

Faculty of Science

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(December-2024)

PRE-SYNOPSIS

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1. INTRODUCTION:

The growing global concern for waste disposal has led to innovative strategies like valuing industrial and agro-waste. One promising approach is using ordered mesoporous silica as a support material for immobilized bio-physico catalysts. This method offers enhanced catalytic performance, improved stability, and reduced environmental footprint. This exploration explores the intricacies of this process.

In this comprehensive exploration, we delve into the intricacies of ordered mesoporous silica synthesis from industrial and agro-waste, the principles of bio-physico catalyst immobilization, and the myriad applications of these immobilized catalysts in diverse physical and chemical processes.

1.1 The Growing Challenge of Waste Disposal: A Deep Dive

The challenges associated with waste disposal extend beyond environmental concerns. The accumulation of waste in landfills and the need for specialized waste management facilities can strain local communities and infrastructure. The costs of waste disposal, including landfill fees, transportation, and treatment expenses, can also impose a significant economic burden on individuals, businesses, and governments.

All the industries throughout the world produces harmful dissipates like agricultural wastes, electronic wastes, industrial wastes etc creating major challenges such as serious health risks and environmental problems[1-4]. As the fused material rises, it cools and solidifies into spherical glassy particles called as fly ash. These fly ash particles create serious ecological problems and also disposal of such a massive quantity of ash has become a vital issue[5-7].

1.2 The Need for Sustainable Solutions

To address the growing challenges of waste disposal, it is imperative to adopt sustainable solutions that minimize environmental impacts and promote resource conservation. These solutions may include:

- **Waste Reduction and Prevention:** Encouraging sustainable consumption patterns, reducing packaging, and promoting recycling and reuse can help to reduce the overall volume of waste generated.
- **Waste Segregation and Recycling:** Implementing effective waste segregation and recycling programs can divert recyclable materials from landfills and recover valuable resources.
- **Composting:** Organic waste can be composted to produce nutrient-rich soil amendments, reducing the amount of waste sent to landfills and promoting sustainable agriculture.
- **Waste-to-Energy:** Converting waste into energy through technologies such as anaerobic digestion and incineration with energy recovery can help to reduce waste volumes and generate renewable energy.

2. REVIEW OF LITERATURE :

In some Previous Years, Green concepts and economic thinking have led to a growing interest in reducing natural waste and industrial hazards, as well as redesigning commercial processes to avoid harmful substances and waste generation. Industrial and agricultural wastes produced after pyrolyzing coal, rice husk, sugar cane husk, or pea nut husk are being used as raw materials and feedstock in further studies.

Coal Fly ash(CFA), Rice husk ash(RHA), Sugar cane husk(SCH) , and Pea nut husk (PNK) are cost-effective, silica-rich raw materials used for creating value-added porous materialslike zeolite molecular sieves[8-12].

Synthetic zeolites made from CFA, RHA, SCH, or PNH can be cost-effective and environmentally friendly solutions for solid acids, adsorbents, molecular sieves, gas separation, purification[13-16], and contaminated waste water[17,18] purification.

Zeolites, aluminosilicate molecular sieves, are microporous inorganic composites with a crystalline structure woven with tetrahedral atoms[19-22] like Al, P, and Si, bridged by oxygen atoms, forming regular channels or cavities.

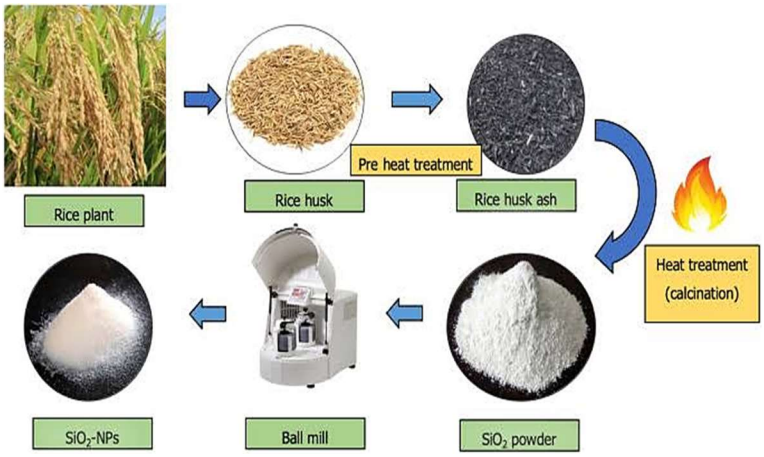

Fig. 1 Protocol for converting RHA to Silica precursor

MCM-41, 48 materials are known for their superior thermal stability, large surface area, and narrow pore size distribution. They are stable under thermal, hydrothermal, mechanical grinding, and acid treatments. These materials have high potential for practical applications, such as adsorbents or mesoporous supports for active catalyst components, especially in the synthesis of fine chemicals involving bulky molecules.

Pore Size (nm)

Microporous : < 200 Mesoporous : <200-5000 Macroporous : > 5000

Table - 1 : Classification of porous materials[20]

Pore diameter	Micropore 2nm mesopores 50nm macropore ^{0}A 1nm 10nm 100nm 1μm
(log scale) Crystal	Zeolites and Related material mesoporous materials
Amorphous material	Pillared clays Porous glasses Silica gels Active carbons Molecular sieving carbons

Zeolites have attracted widespread attention in scientific field as well as in industrial applications. It is a dynamic and rapidly growing field involving different branches of science like crystallography, theoretical physics, catalysis, inorganic, organic, physical, colloidal biochemistry, environmental science, mineralogy, geology and oceanography.

Mesoporous materials, with their wide pore diameter, are easily accessible to large molecules and are crucial for processing. The formation of these materials is facilitated by the "liquid-crystal templating" (LCT) mechanism, which allows for the formation of various crystallographically well-defined frameworks. Their large regular pore structure makes them ideal for liquid-phase acidic and basic catalysis, allowing rapid diffusion of reactants and products through pores.

Various synthesis strategies have been used to prepare mesoporous molecular sieves, which are then filtered, washed, and dried. The mesoporous silicate/aluminosilicate network is obtained by calcination, removing surfactant molecules.

2.1 SILICA AND THEIR STRUCTURES :

Post-synthesis immobilization of metals and organometallic complexes on the surface of mesoporous silicates allows for the preparation of multifunctional molecular sieves with desired catalytic properties.

Mesoporosity and high surface area of mesoporous materials have been widely used for immobilizing various metals and bulky organometallic complexes. The immobilization of metal complexes is mostly done by ion exchange followed by complexation with ligand and direct incorporation.

Inorganic-organic hybrid materials offer advantages due to their mechanical, thermal, or structural stability, while organic features are more readily modified for specific applications in catalysis, separation, or sensing. Organically functionalized mesoporous silicas can be prepared at room temperature or higher temperature by co-condensation of tetra alkoxysilane in the presence of surfactant and auxiliary chemicals.

Organo functional groups can also be introduced to the pore surface of mesoporous silica as terminal groups for organic monolayers by post-synthesis modification. Extensive research has been conducted to control crystal size, pore dimensions, and pore sizes, particularly for MCM-41, 48 with unidirectional channels.

High-quality hexagonal mesoporous materials with good thermal stability and smaller crystal sizes can be obtained by heating precursor gels in the microwave at 150ºC for 1 hour or less.

APPLICATIONS OF MATERIAL IN INDUSTRIAL USAGE

Purification And Treatment of Water from Agro Industrial :

In industrial use of various materials of Mesoporous materials are highly effective sorbents for various water treatment applications, including water softening, heavy metal removal, phosphate and phenol removal, dissolved organic and inorganic compound removal, solid impurity separation, and radioactive wastewater purification, among others.

Public water must be potable, suitable for drinking and agriculture, and may contain various organic and inorganic impurities. These can be separated using mesoporous zeolites, a special class of inorganic compounds, which can help separate out impurities.

Among the various types of purification methods, ion exchange methodology is the cost effective methodology and is one of the most well-developed, common, and effective treatment methods for treatment of contaminated water.

3. OBJECTIVES OF THE RESEARCH / PROPOSED HYPOTHESIS

The objectives of the present work are:

1. To study the effect on the adsorptive behavior of pre and post modified synthesized materials
2. Post Modification of synthesized materials (metal incorporated synthesized materials).
3. To synthesize MCM-41, 48 using silica extracted from CFA and RHA, CSH, PNH and to optimize the synthesis procedures.
4. To study the effect on the adsorptive behavior of pre and post modified synthesized materials
5. To deal with an account of conductivity behavior of selected samples
6. To study the catalytic activity of synthesized materials in diverse reactions.
7. To study the kinetics of crystallization on synthesized materials such as
    - Effect of crystallization time
    - Effect of Temperature
    - Effect of P^H
    - Effect of calcination period
    - Effect of NaOH

4. METHODOLOGY TO BE ADOPTED:

Using an ion exchange approach for post modification, heterogeneous chemical processes, and "Freundlich adsorption" applications, the hydrothermal synthesis of mesoporous molecular sieves was carried out in a hydrothermal reactor[23-25] under stirring circumstances.

Consider general factors like material composition, processing methods, and desired properties. Computational methods, can be used for predicting material properties before experimental trials.Propose material modifications or innovations.

For example, adjusting the composition of materials, altering manufacturing processes (like additive manufacturing), or introducing nanostructures.

5. SIGNIFICANCE OF STUDY:

Environmental remediation efforts often face challenges due to the high costs of synthesising mesoporous materials from chemical sources like silica and alumina. However, researchers are exploring cheaper raw materials like industrial waste CFA and agro waste rice husk for their potential applications in water purification technology, industrial catalysts, and molecular sieves.

Extracting silica from silica-rich materials like CFA, RHA, CSH, and PNH has led to the synthesis of mesoporous molecular sieves. However, the amorphous framework structure and lack of active centres in pure silica mesoporous materials limit their direct application.

Modifying these materials with heteroatoms like Al and Cs has enhanced catalytic and adsorption activity. This study aims to prepare optimized mesoporous, functional materials for selective catalytic transformation of organic molecules under ambient conditions.

6. PROPOSED WORK PLAN:

The study aims to synthesize silica-based mesoporous materials from industrial and agricultural wastes and their applications in various physical and chemical perspectives over three years.

First Year:-

1. The objective is to gather references from relevant fields.
2. Attend relevant seminars, conferences, or workshops.
3. The study aims to analyze various papers and determine the most suitable synthesis scheme, either through hydrothermal or in situ methods.

Second Year:-

1. To study aims to explore both existing and new techniques to be utilized in our research work.
2. The process involves collecting industrial and agricultural wastes like coal fly ash, rise husk ash, pea nut husk, and cane sugar husk, and calcinating them to remove carbonius and magnetic material through magnetic stirring.
3. The raw material, which has been calcined and demagnetized, will be screened to obtain a fine residue for further investigation.
4. Some characterization such as XRD, FTIR, SEM and EDAX will be taken to find out the percentage of silica and other components.
5. The selection of rich silica raw material will be made for the further synthesis of mesoporous silicious material.
6. The leaching process will extract silica from sodium hydroxide, resulting in a supernatant for synthesis purposes.
7. The objective is to synthesize MCM-41, 48 from various mesoporous materials.
8. Synthesis process will be optimized by studying the effect of synthesis temperature, calcinations temperature, pH, time, effect of NaOH, reusability, etc.
9. Post modified synthesized material will be used in some physical and chemical applications.

Third Year:-

1. The goal is to publish the work in reputable, peer-reviewed international journals.
2. The task involves gathering data for thesis writing, which will be submitted to the appropriate authority.

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